Ülesanne 8

TMR-katseklaasis segati kokku väike kogus (2-10 µL) erinevaid puhtaid orgaanilisi lahusteid (alla 10) ja lisati 0,6 mL deutereeritud kloroformi (99,8% deuteeriumrikastusega $CDCl_3$).

Mõõdeti ^{1}H (700,1 MHz) ja $^{13}C\{^{1}H\}$ ning APT (176,0 MHz) TMR spektrid. Need spektrid ja suurendused nendest on toodud järgnevatel lehtedel.

Raportisse kirjutada:

1. Kasutage viidetes 1 või 2 toodud tabeleid või muid saadaolevaid tabeleid ning leidke, mis orgaanilisi lahusteid kokku segati. Nimekiri esitage tabeli kujul:

Ühendi nimetus	Ühendile kuuluvad ^{1}H TMR signaalide keemilised nihked	Ühendile kuuluvad $^{13}C\{^{1}H\}$ TMR signaalide keemilised nihked
Trikloro(^{2}H)metaan	–	77,2 ppm
jne.		

NB! Arvestage, et tabelis olevad puhaste ainete keemiliste nihete väärtused on veidi erinevad reaalselt solventide segus mõõdetud väärtustest. Samuti pange tähele, et spektris peavad olemas olema kõikide ühe molekuli fragmentide signaalid ning ka signaalide multipletsused peavad klappima.

2. Hinnake 2 tuvastatud solvendi (valige välja 2 solventi, mille signaalid spektritelt tuvastanud olete) omavahelist suhtelist sisaldust (moolide järgi). Näidake ära lahenduskäik.

3. Mõõtke ära ^{1}H TMR spektris 1,21 ppm juures asuva tripleti sidestuskonstandi suurus (hertsides ja täpsusega 1 koht pärast koma).

Viited:

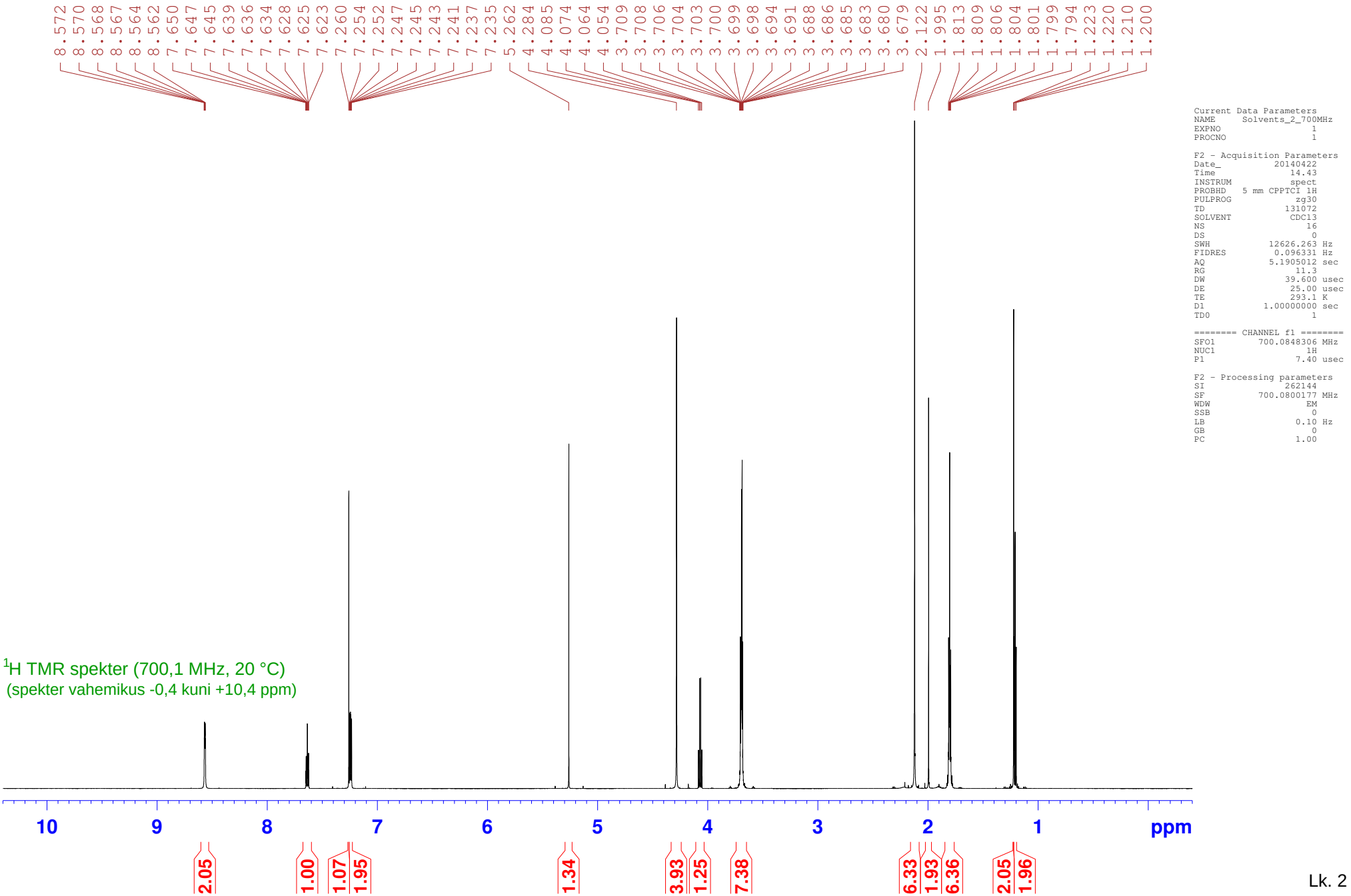
1. Fulmer, G.R.; Miller, A.J.M.; Sherden, N.H.; Gottlieb, H.E.; Nudelman, A.; Stoltz, B.M.; Bercaw, J.E.; Goldberg, K.I. NMR Chemical Shifts of Trace Impurities: Common Laboratory Solvents, Organics, and Gases in Deuterated Solvents Relevant to the Organometallic Chemist. *Organometallics* **2010**, *29*, 2176-2179. **DOI:** 10.1021/om100106e
2. Gottlieb, H. E.; Kotlyar, V.; Nudelman, A. NMR Chemical Shifts of Common Laboratory Solvents as Trace Impurities. *J. Org. Chem.* **1997**, *62*, 7512-7515. **DOI:** 10.1021/jo971176v

Ülesanne on kättesaadav PDF-formaadis aadressilt: http://kodu.ut.ee/~laurit/AK2/

Raportit soovin saada oma e-posti aadressile lauri.toom@ut.ee ainult PDF-formaadis. Raporti 1. versiooni esitamise tähtaeg on 15. mai 2015 kell 17:00. Minupoolse tagasiside põhjal korrektselt parandatud lõpp-versiooni esitamise tähtaeg on 29. mai 2015 kell 17:00.

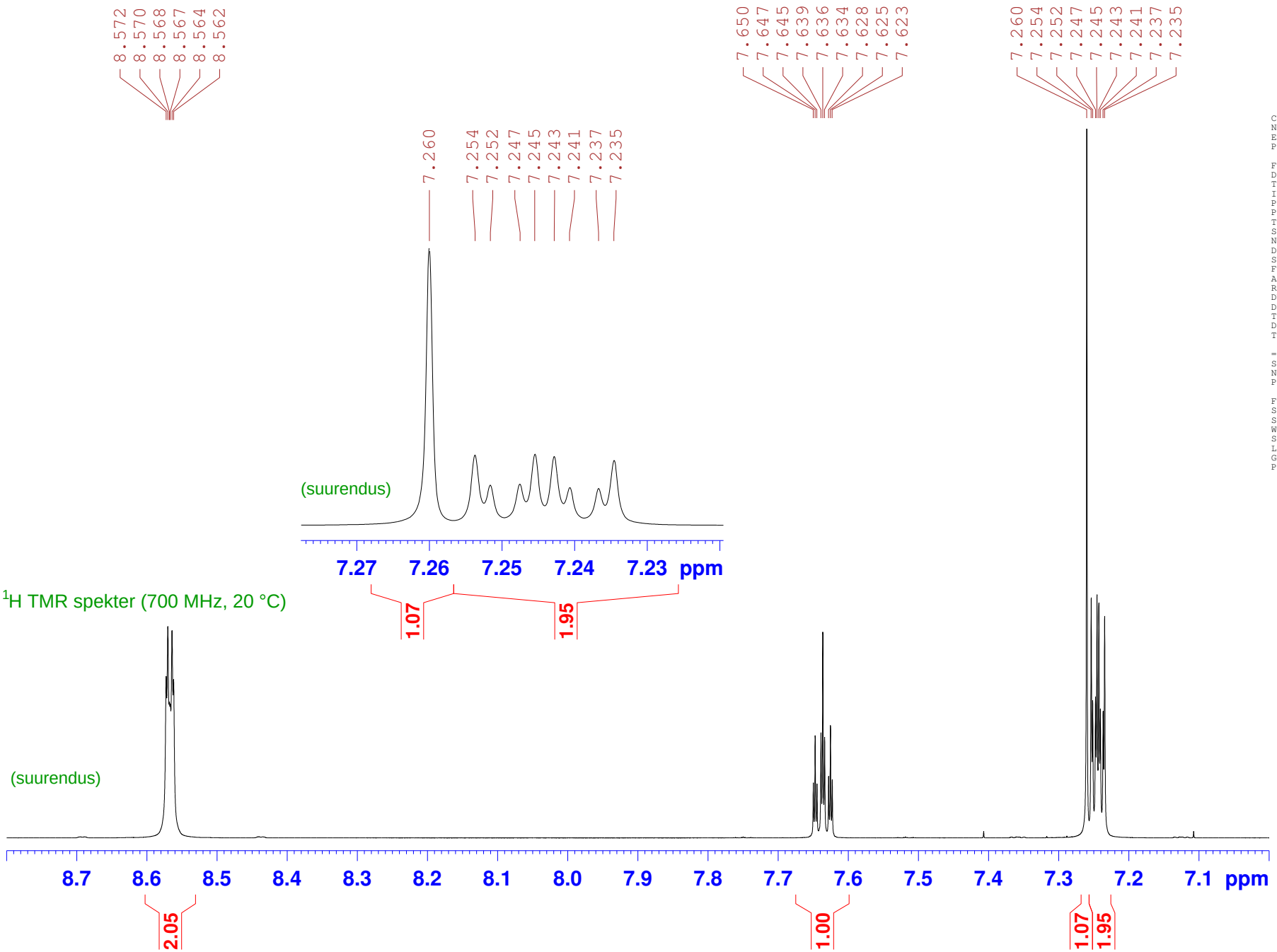
Lauri Toom
lauri.toom@ut.ee

UNIVERSITY OF TARTU



Lauri Toom
lauri.toom@ut.ee

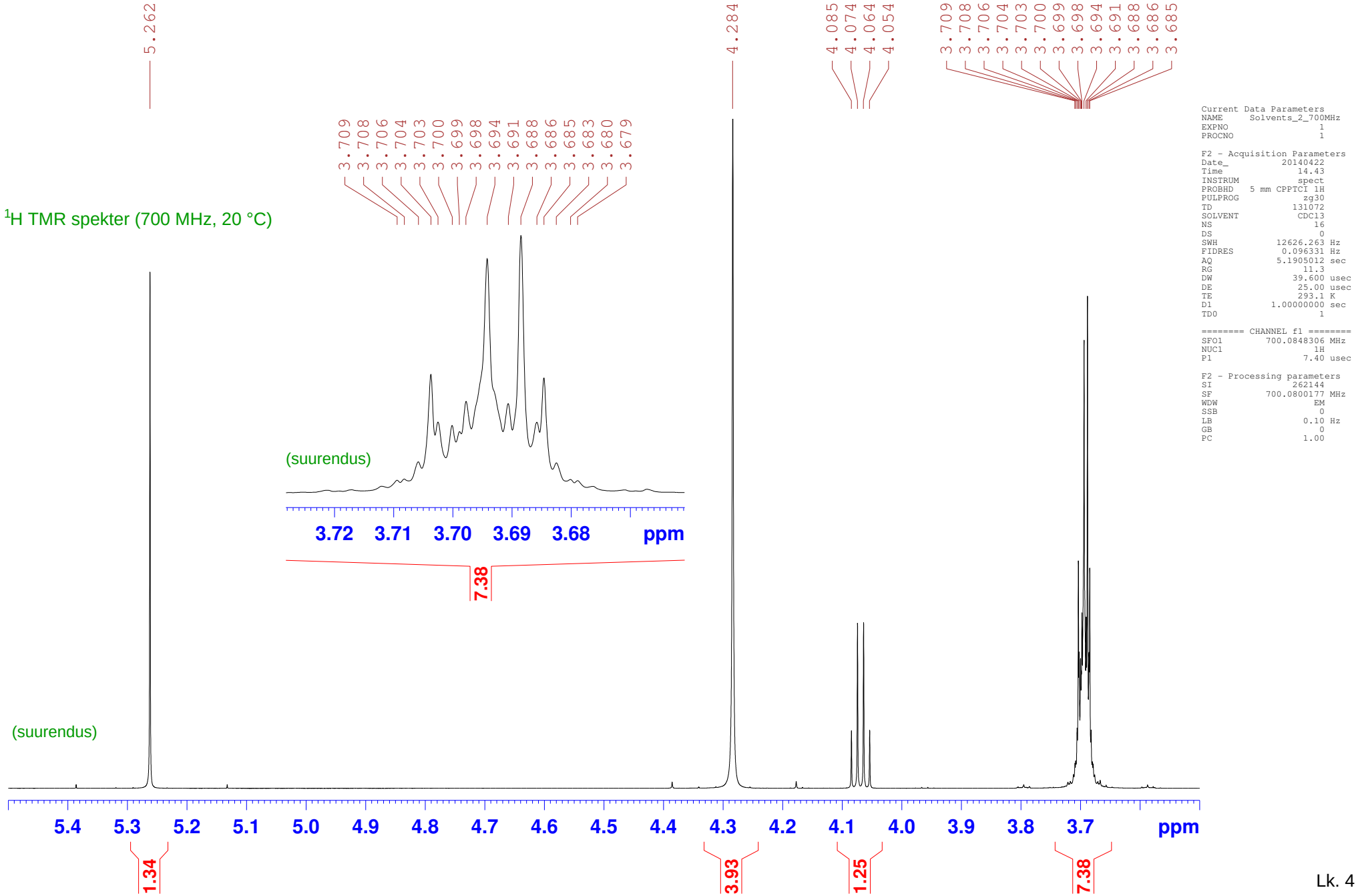
^{1}H TMR spekter (700 MHz, 20 °C)




```
Current Data Parameters
NAME      Solvents_2_700MHz
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_           20140422
Time              14.43
INSTRUM           spect
PROBHD   5 mm CPPTCI 1H
PULPROG           zg30
TD              131072
SOLVENT           CDCl3
NS                  16
DS                   0
SWH           12626.263 Hz
FIDRES         0.096331 Hz
AQ             5.1905012 sec
RG                11.3
DW              39.600 usec
DE              25.00 usec
TE              293.1 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         700.0848306 MHz
NUC1               1H
P1               7.40 usec

F2 - Processing parameters
SI              262144
SF          700.0800177 MHz
WDW               EM
SSB                0
LB              0.10 Hz
GB                 0
PC              1.00
```

Lauri Toom
lauri.toom@ut.ee

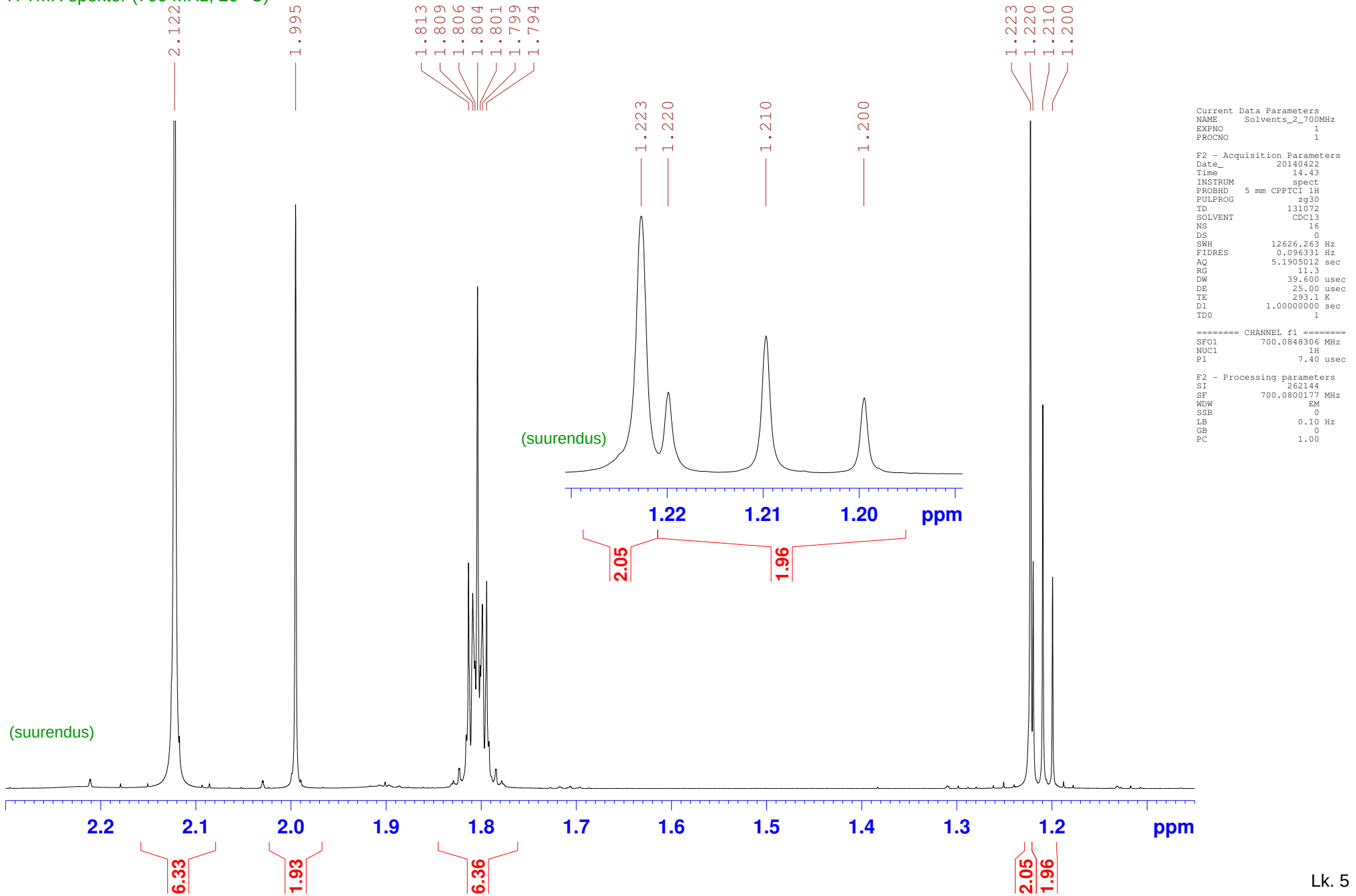
^{1}H TMR spekter (700 MHz, 20 °C)




```
Current Data Parameters
NAME      Solvents_2_700MHz
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140422
Time              14.43
INSTRUM           spect
PROBHD   5 mm CPPTCI 1H
PULPROG             zg30
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           12626.263 Hz
FIDRES         0.096331 Hz
AQ             5.1905012 sec
RG                 11.3
DW                39.600 usec
DE                25.00 usec
TE                293.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         700.0848306 MHz
NUC1                1H
P1                 7.40 usec

F2 - Processing parameters
SI                262144
SF          700.0800177 MHz
WDW                   EM
SSB                    0
LB                 0.10 Hz
GB                     0
PC                 1.00
```

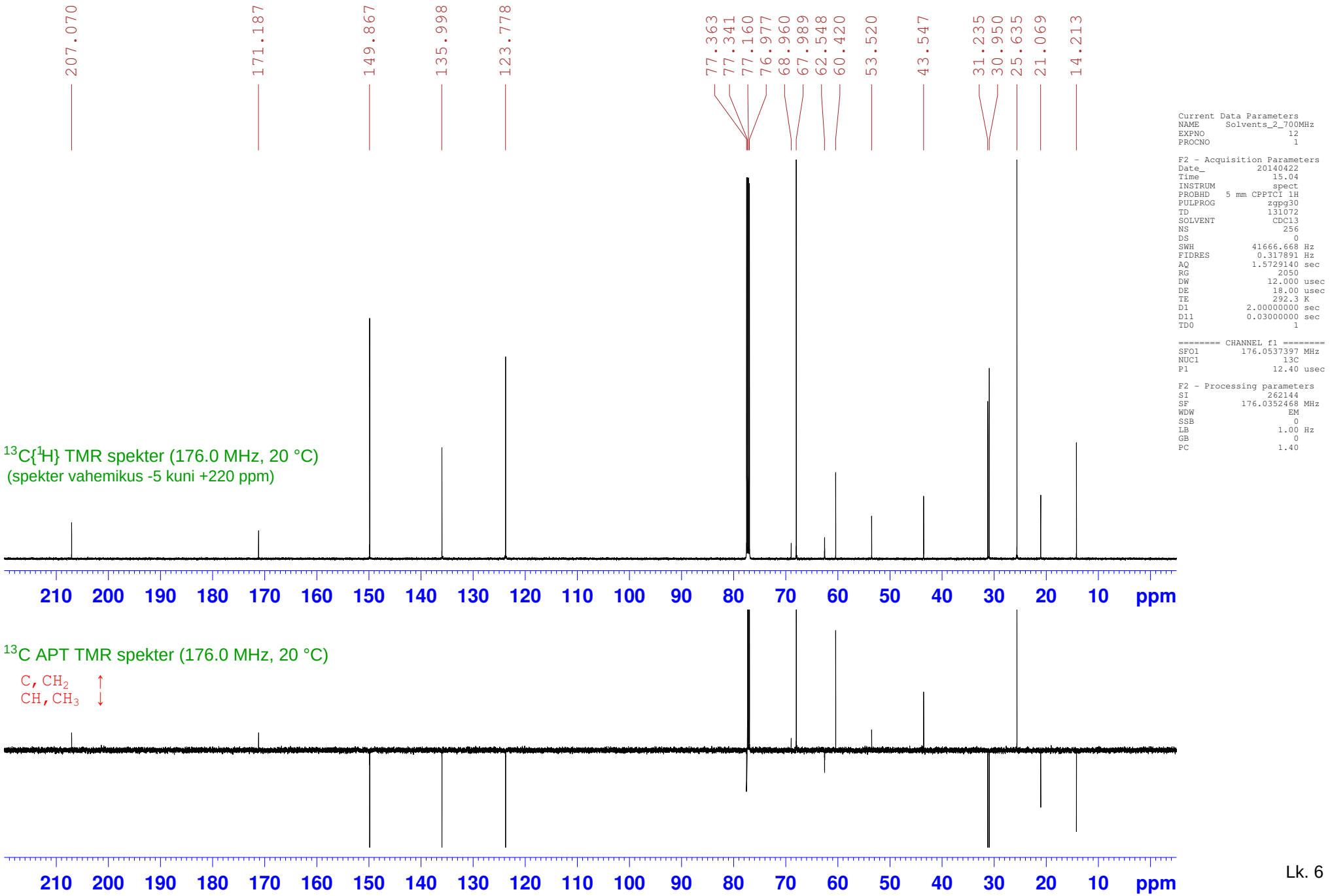
Lauri Toom
lauri.toom@ut.ee

^{1}H TMR spekter (700 MHz, 20 °C)

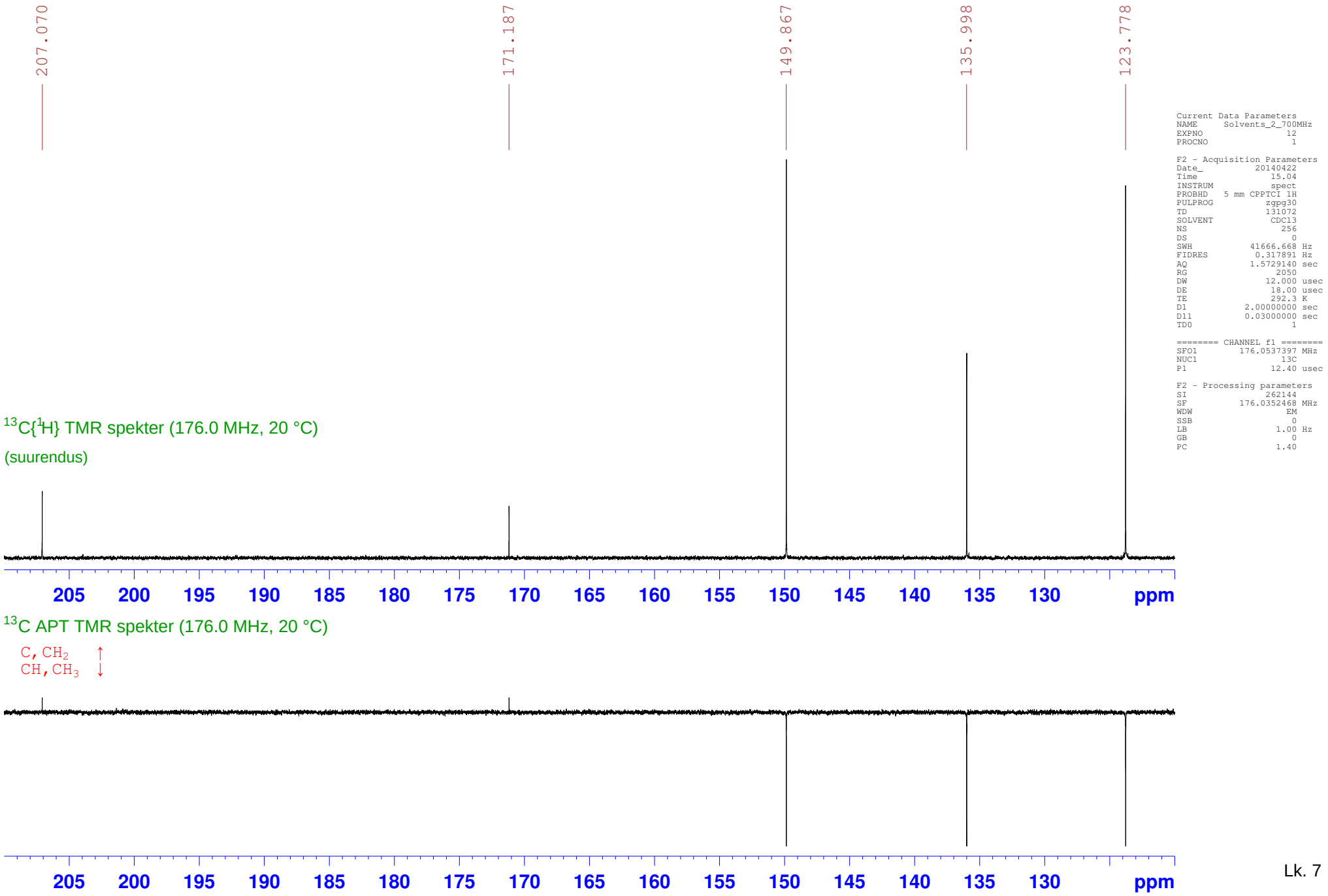



```
Current Data Parameters
NAME      Solvents_2_700MHz
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_           20140422
Time              14.43
INSTRUM           spect
PROBHD   5 mm CPPTCI 1H
PULPROG           zg30
TD              131072
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12626.263 Hz
FIDRES        0.096331 Hz
AQ            5.1905012 sec
RG                 11.3
DW               39.600 usec
DE               25.00 usec
TE               293.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         700.0848306 MHz
NUC1               1H
P1                7.40 usec

F2 - Processing parameters
SI              262144
SF          700.0800177 MHz
WDW               EM
SSB                0
LB              0.10 Hz
GB                 0
PC              1.00
```

Lauri Toom
lauri.toom@ut.ee



Lauri Toom
lauri.toom@ut.ee

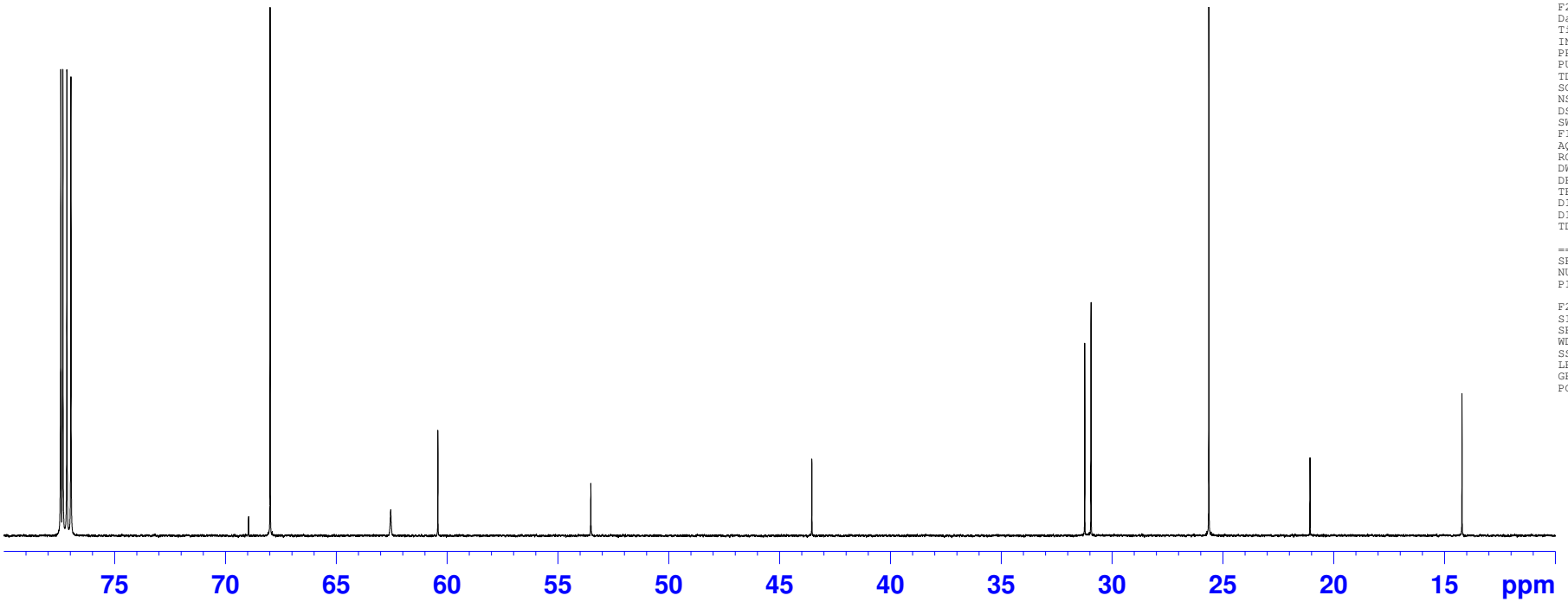


Lauri Toom
lauri.toom@ut.ee

$^{13}C\{^{1}H\}$ TMR spekter (176.0 MHz, 20 °C)

(suurendus)

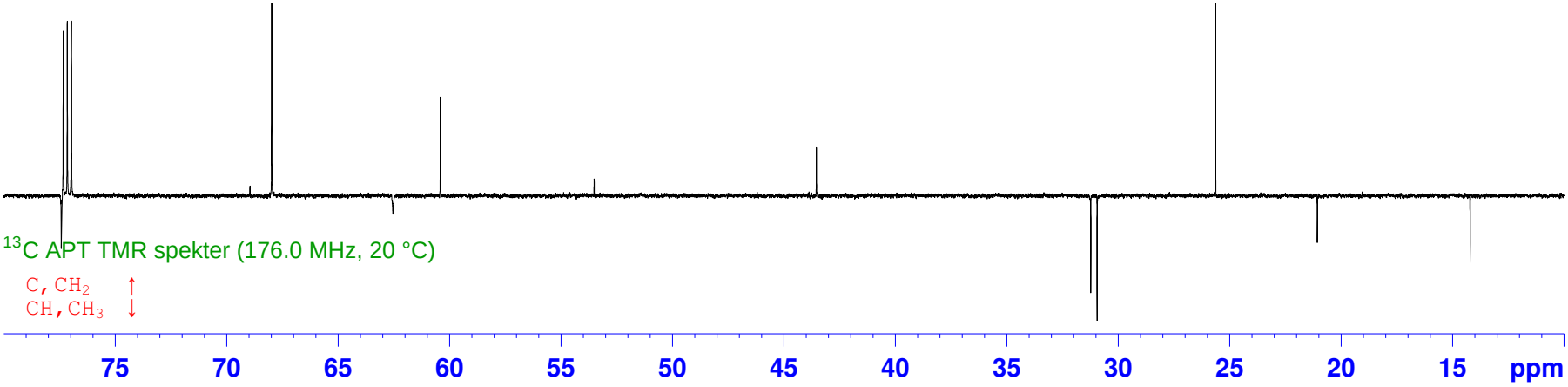
77.363 77.341 77.160 76.977 68.960 67.989 62.548 60.420 53.520 43.547 31.235 30.950 25.635 21.069 14.213




```
Current Data Parameters
NAME      Solvents_2_700MHz
EXPNO                    12
PROCNO                    1

F2 - Acquisition Parameters
Date_              20140422
Time                 15.04
INSTRUM              spect
PROBHD   5 mm CPPTCI 1H
PULPROG            zgpg30
TD                 131072
SOLVENT              CDCl3
NS                     256
DS                       0
SWH             41666.668 Hz
FIDRES          0.317891 Hz
AQ              1.5729140 sec
RG                    2050
DW                12.000 usec
DE                18.00 usec
TE                 292.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          176.0537397 MHz
NUC1                    13C
P1                  12.40 usec

F2 - Processing parameters
SI                  262144
SF            176.0352468 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

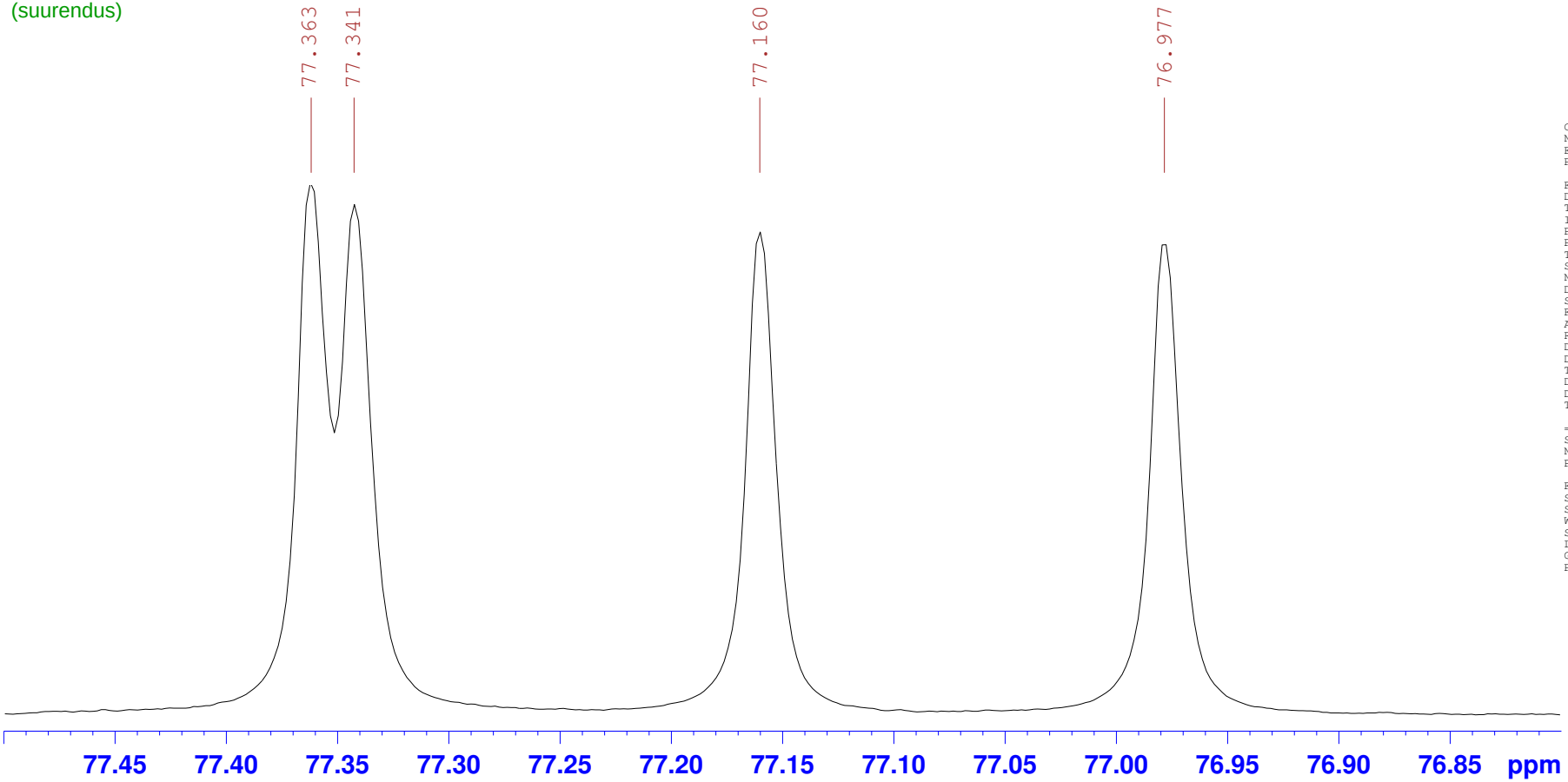
^{13}C APT TMR spekter (176.0 MHz, 20 °C)

C, CH_2 ↑
CH, CH_3 ↓



Lauri Toom
lauri.toom@ut.ee

$^{13}C\{^{1}H\}$ TMR spekter (176.0 MHz, 20 °C)
(suurendus)



Current Data Parameters
NAME Solvents_2_700MHz
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140422
Time 15.04
INSTRUM spect
PROBHD 5 mm CPPTCI 1H
PULPROG zgpg30
TD 131072
SOLVENT CDC13
NS 256
DS 0
SWH 41666.668 Hz
FIDRES 0.317891 Hz
AQ 1.5729140 sec
RG 2050
DW 12.000 usec
DE 18.00 usec
TE 292.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 176.0537397 MHz
NUC1 13C
P1 12.40 usec

F2 - Processing parameters
SI 262144
SF 176.0352468 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{13}C APT TMR spekter (176.0 MHz, 20 °C)